IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF:
Shigeki NAKATSUKASA, Hiroyuki OHGI and Kazuyori YOSHIMI
: GROUP ART UNIT: 1752
SERIAL NO: 08/974,490 : EXAMINER: Barbara Gilmore
FILED: November 19, 1997

FOR: Resin composition and multilayered structure

DECLARATION UNDER 37 C.F.R. § 1.132

Honorable Commissioner of Patents and Trademrks,
Washington, D.C. 20231

Sir:

I, Kazuyori Yoshimi, residing at 1621, Sakazu, Kurashiki-city, Okayama Prefecture, Japan do hereby declare as follows:

I have graduated Okayama University, Department of Science, Master Course in March, 1970; and from April, 1970 up to date I have been an employee of KURARAY CO., LTD. and have been mainly engaged in the field of development of ethylene-vinyl alcohol copolymer (hereinafter referred to as EVOH). I am familiar with the instant technical field. And I am a third inventor of the present application.

I declare that the results of experiment I have conducted are as follows and that they are true to the best of my knowledge and belief.

In order to show flexural fatigue resistance and impact resistance of the resin composition comprising EVOH and high density polyethylene (HDPE), experiment has been carried out under my supervision as follows.

Experiment 1

A dry blend was prepared from the following two components.

(A) High density polyethylene, "HI-ZEX 8200B" from Mitsui Chemicals, Inc.
(10 parts by weight)
Density = 0.956 g/cm^3
MFR = 0.03 g/10 min (190℃, 2,160 g load)
Ultimate Tensile (ASTM D-638) = 40 MPa

(B) EVOH (90 parts by weight)
Ethylene content = 32 mol%
Degree of hydrolysis = 99.6%
MFR = 3.1 g/10 min (210℃, 2,160 g load)
Content of phosphorus compound (potassium dihydrogenphosphate) = 100 ppm (in terms of phosphorus element)
Content of potassium = 125 ppm (in terms of potassium element)
Content of sodium salt (sodium acetate) = 65 ppm (in terms of sodium element)

The dry blend was pelletized by extrusion through an extruder (40 mm in diameter, L/D = 24, compression ratio = 3.8) having a Madock-type mixing zone. Thus there was obtained the resin composition.

A 5-layered film specified below was produced by co-extrusion from the resin composition and the following two components fed into separate extruders.

Layer construction:

LLDPE/AD/RC/AD/LLDPE = 50/5/20/5/50 μm

Total thickness = 130 μm

• LLDPE: Ethylene-α-olefin copolymer produced by using a conventional Ziegler catalyst ("UF420" from Mitsubishi Chemical Corporation)

density = 0.925 g/cm^3

MFR = 0.8 g/10 min (210℃, 2,160 g load)

• AD: Linear low-density polyethylene graft-modified with maleic anhydride for the adhesive resin layer ("Admer NF500" from Mitsui Chemicals, Inc.)

MFR = 3.6 g/10 min (210℃, 2,160 g load)

• RC: Resin composition

Each component was extruded under the following conditions.

• LLDPE: at 200 to 240℃ through a 65-mm single-screw extruder with an L/D ratio of 22.

• AD: at 160 to 220℃ through a 40-mm single-screw extruder with an L/D ratio of 26.

• Resin composition: at 200 to 240℃ through a 40-mm single-screw extruder with an L/D ratio of 26.

The melt was discharged from a feed block die (600 mm wide) at 240℃.

A specimen (12 x 8 inches) was cut out of the resulting multilayered film and conditioned at 20℃ and 65 %RH. The specimen was made into a cylinder, 3.5 inches in diameter. The cylinder was held by grips at its ends on a Gelbo Flex Tester (made by Rigaku Kogyo). Initially, the grips were 7 inches apart. The specimen was twisted by turning the grips in opposite direction through an angle of 440° such that the distance between the grips was decreased to 3.5 inches. Then the grips were moved straight and horizontally so that the distance between them

was decreased to 2.5 inches. Finally, the grips were returned to their original positions. (When the specimen was flexed to the utmost limit, the grips were 1 inch apart.) These steps were repeated at a rate of 40 cycles per minute. This test was carried out at 20℃ and 65 %RH. Pinholes appeared in the specimen when the specimen was observed after 500 cycles.

A sample of flexible container was prepared by heat sealing from the multilayered film. (Two sheets of film measuring 20 by 30 cm were placed one over the other and three sides were heat-sealed, with the 20-cm side left open.) This sample was filled with water (20℃) and the open side was heat-sealed. In this way there was obtained a flexible container for bag-in-box. This container was dropped on a concrete floor. The height for the container to break (allowing water to leak) was recorded. This test was repeated for 30 samples and the results were calculated according to JIS K7211 (Section 8 for calculations) to obtain the height for 50% of the samples to break. The height for the container to break was 1.1 m.

I, the undersigned declarant, declare further that all statement made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and; further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001, of Title 18, of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this 26 th day of December, 2001

Name:

Kazuyori Yoshimi

Kazuyori Yoshimi

ENGAGE

ENGAGE EG 8100

Polyolefin Elastomer for General Purpose Elastomeric Applications

- General purpose elastomer
- Excellent impact properties in blends with polypropylene and polyethylene
- Excellent flow characteristics
- Excellent heat aging and compression set when crosslinked

ENGAGE* EG 8100 polyolefin elastomer is a saturated ethylene-octene copolymer that provides excellent flow properties and is efficiently crosslinked by peroxide, silane or irradiation. ENGAGE EG 8100 POE delivers exceptional heat aging, compression set, and weather resistance properties. This elastomer is also highly effective as an impact modifier in polyolefins.

Physical Properties	Test Method	Values[1]
Density, gm/cc	ASTM D-792	.87
Mooney Viscosity, ML 1+4 @ 121°C	ASTM D-1646	23
Percent Comonomer, octene	Dow[2]	24
Melt Index, dg/min	ASTM D-1238	1
Melt Flow Ratio, I_{10}/I_2	ASTM D-1238	7.5
Dow Rheology Index (DRI)	Dow[3]	2
Ultimate Tensile, psi (MPa)	ASTM D-638	1,500 (10.3)
100% Tensile Modulus, psi (MPa)	ASTM D-638	350 (2.4)
Ultimate Elongation, %	ASTM D-638	800
Hardness, Shore A	ASTM D-2240	75
Brittleness Temperature, °C	ASTM D-746	<-76
Flexural Modulus, 2% Secant, psi (MPa)	ASTM D-790	2,340 (16.1)

*Trademark of The Dow Chemical Company.

[1]These are typical properties only, and are not to be regarded as sales specifications.

[2]Based on ASTM D-2238, Method B.

[3]A calculated value based on complex viscosity that expresses the relative influence of long chain branching on the extrudability of homogeneous (single site catalyst) polyolefins.

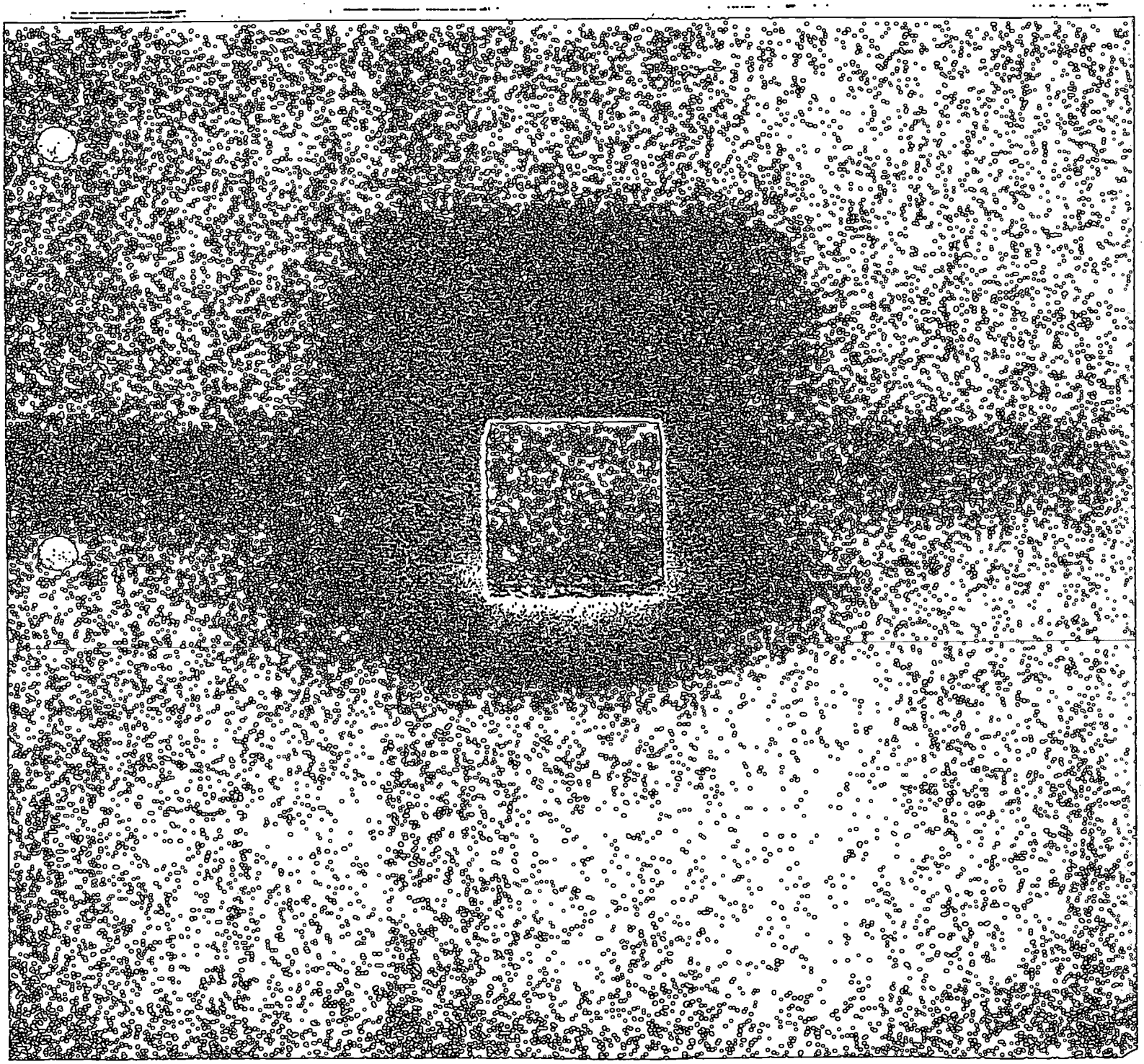
— See "Handling Considerations" on the reverse side.

DOW Dow Plastics

三井化学

人々の暮らしを豊かに彩る ポリエチレン

POLYETHYLENE



HI-ZEX
ハイゼックス

高密度ポリエチレン

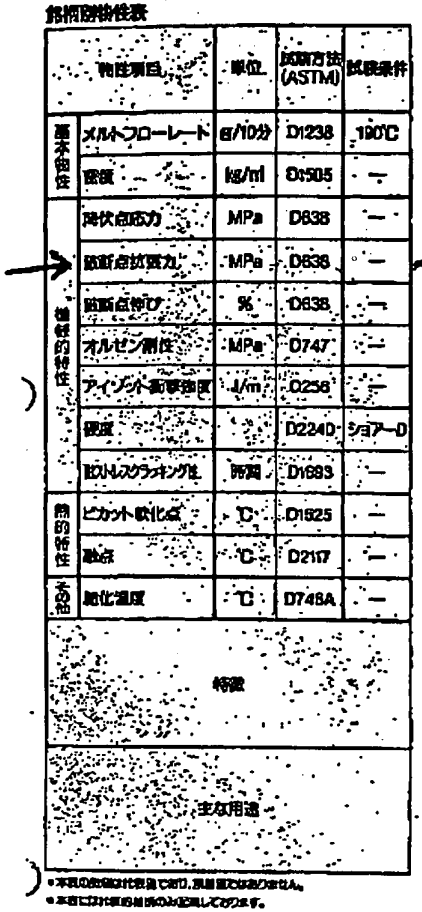
銘柄別物性表

	物性項目	単位	試験方法(ASTM)	試験条件	射出成形					中空成形						押出成形								フィルム		
					1300J	2100J	2100JH	2200J	2208J	3000B	6100B	6202B	6008B	6200B	8200B	6000S	6000SR	5000SF	6000H	6300M	6305E	7500M	7800M	3300F	7000F	8000F
基本物性	メルトフローレート	g/10分	D1238	190℃	13	6	9	5.2	5.2	0.62	0.26	0.33	0.38	0.36	0.03	0.8	0.35	0.65	0.11	0.11	0.8	0.03	0.04	1.1	0.04	0.03
基本物性	密度	kg/m³	D1505	—	965	958	956	968	968	964	947	964	958	958	956	954	958	959	960	952	953	961	959	954	956	950
機械的特性	降伏点応力	MPa	D638	—	30	26	26	32	32	30	23	29	28	28	29	25	27	28	27	22	24	30	29	25	23	21
機械的特性	破断点抗張力	MPa	D638	—	15	20	15	17	17	25	33	37	32	32	40	35	35	25	30	30	23	32	33	35	30	35
機械的特性	破断点伸び	%	D638	—	>300	>500	>300	>300	>500	>500	>500	>500	>500	>500	>500	>500	>500	>500	>500	>500	>500	>500	>500	>500	>500	>500
機械的特性	オルゼン剛性	MPa	D747	—	1200	1000	1000	1300	1300	1200	700	1200	1100	1100	1000	900	800	1000	1000	800	800	1100	1000	800	1000	800
機械的特性	アイゾット衝撃強度	J/m	D256	—	30	40	40	50	50	100	200	NB	120	120	NB	200	100	80	150	120	150	NB	NB	200	NB	NB
機械的特性	硬度		D2240	ショアーD	65	63	62	65	65	65	61	64	64	64	63	63	64	65	64	62	62	66	65	62	64	61
機械的特性	耐ストレスクラッキング性	時間	D1693	—	3	8	15	4	4	24	>600	40	300	300	>600	30	150	100	>600	>600	>600	>600	>600	90	>600	>600
熱的特性	ビカット軟化点	℃	D1525	—	128	121	122	130	130	128	119	128	123	123	123	123	124	124	125	122	122	125	124	123	124	122
熱的特性	融点	℃	D2117	—	134	131	131	135	135	134	128	134	132	132	132	131	132	132	132	131	130	133	132	131	131	130
その他	脆化温度	℃	D746A	—	<-60	<-60	<-60	<-60	<-60	<-60	<-60	<-60	<-60	<-60	<-60	<-60	<-60	<-60	<-60	<-60	<-60	<-60	<-60	<-60	<-60	<-60
特徴					流動性	機械強度	耐ストレスクラッキング性	剛性	剛性	[illegible]	耐ストレスクラッキング性	衝撃強度	耐ストレスクラッキング性	耐ストレスクラッキング性	衝撃強度	延伸性	紡糸性	成形性	剛性	耐薬品性	高速成形性	耐圧性	高衝撃性	剛性	衝撃強度	超衝撃強度
特徴					剛性		外観		耐候性		スクイズ性		[illegible]			耐候性	耐候性	機械強度		耐ストレスクラッキング性	耐ストレスクラッキング性	耐ストレスクラッキング性	耐ストレスクラッキング性			
特徴																	結節強度			クリープ性		成形性				
主な用途					日用品	工業部品	工業部品	工業部品	コンテナ	食品容器	化粧品容器	灯油缶	洗剤容器	洗剤容器	大型容器	ネット	クロス	結束テープ	シート	一般用水道管	電線被覆	一般リブ付管	高耐圧リブ付管	プロテクトフィルム	[illegible]強化フィルム	[illegible]強化フィルム
主な用途					工業部品	キャップ		日用品	パレット	工業部品	医薬容器	工業薬品缶	シャンプー容器	シャンプー容器	工業薬品缶	ロープ	ネット	一般フィルム	工業部品	電設管				ひねり包装		
主な用途									日用品		シャンプー容器		化粧品容器	化粧品容器		結束テープ	結束テープ			上水道管						

●本表の数値は代表値であり、規格値ではありません。
●本表に記した用途は代表例のみ記載しております。

単位換算
密度 1g/cm³=1000kg/m³
降伏点応力 1kg/cm²=0.098MPa
破断点抗張力 1kg/cm²=0.098MPa
オルゼン剛性 1kg/cm²=0.098MPa
アイゾット衝撃強度 1kg/cm=9.807J/m

Partial English translation f Catalog of "HI-ZEX" (Mitsui Kagaku)



特性項目		単位	試験方法 (ASTM)	試験条件
基本特性	メルトフローレート	g/10分	D1238	190℃
	密度	kg/m³	D1505	—
機械的特性	降伏点応力	MPa	D638	—
	破断点抗張力	MPa	D638	—
	破断点伸び	%	D638	—
	オルゼン剛性	MPa	D747	—
	アイゾット衝撃強度	J/m	D256	—
	硬度		D2240	ショアーD
	耐ストレスクラッキング性	時間	D1693	—
熱的特性	ビカット軟化点	℃	D1525	—
	融点	℃	D2117	—
その他	脆化温度	℃	D746A	—
特徴				
主な用途				

Properties		unit	test method (ASTM)	test conditions
basic properties	Melt flow rate	g / 10 min.	D1238	190℃
	density	kg / m³	D1505	—
mechanical character-istic	Stress at yield	MPa	D638	—
	Ultimate Tensile	MPa	D638	—
	Ultimate Elongation	%	D638	—
	Olsen hardness	MPa	D747	—
	Izod impact Strength	J / m	D256	—
	Hardness		D2240	Shore D
	stress crack resistance	hour	D1693	—
thermal character-istic	Vicat softening temperature	℃	D1525	—
	melting point	℃	D2117	—
others	Breittleness Temperature	℃	D746A	—
characteristic				
main use				

VERIFICATION OF TRANSLATION

I, Takahiro Sekiguchi of c/o Kuraray Co., Ltd. 1621, Sakazu, Kurashiki-City, Okayama-Pref., Japan

declare as follows:

1. That I am well acquainted with both the English and Japanese languages, and

2. That the attached document is a true and correct translation made by me to the best of my knowledge and belief of:

   a part of catalog of HI-ZEX (Mitsui Chemical Corporation).

December 27, 2001
(Date)

Takahiro Sekiguchi
(Signature of Translator)